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QUANTITATIVE DETERMINATION OF CORROSION INHIBITOR
LEVELS IN JET FUELS BY HPLC(U) SOUTHEASTERN
MASSACHUSETTS UNIV NORTH DARTMOUTH N A WECHTER

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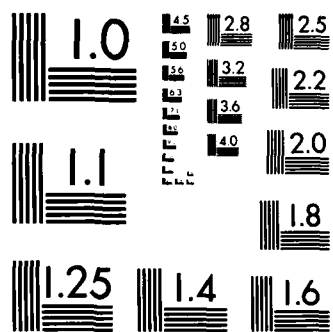
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"QUANTITATIVE DETERMINATION OF CORROSION INHIBITOR LEVELS IN JET FUELS BY HPLC"

SUBMITTED TO DR. DENNIS HARDY, NAVAL RESEARCH LABORATORY

BY DR. MARGARET A. WECHTER, SOUTHEASTERN MASSACHUSETTS UNIVERSITY

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INTRODUCTION

Corrosion inhibitors are mandatory additives which are added to Navy jet fuels to reduce corrosion of transfer lines and storage tanks. In addition, they are known to enhance lubricity properties of hydro-treated fuels. It is known that, as fuels pass through transfer lines or are stored, the level of corrosion inhibitor present decreases and with this decrease there is a corresponding decrease in both the corrosion protection and the lubricity characteristics of the fuel. Since there are potentially deleterious effects resulting from a decrease in corrosion inhibitor concentrations, a method for quantifying their concentrations was desired in order to be able to make appropriate after market adjustments to fuels. Thus, the ultimate goal of this project was to develop an analytical method to quantitatively determine the amount of corrosion inhibitor present in fuel samples. A secondary goal was to gain more specific experimental evidence regarding the fate of the inhibitors.

because the typical additive concentration is generally less than 35 ppm (W/V), the concentration range 2-30 ppm was chosen. The method of choice was gel permeation chromatography (gpc) combined with a solvent extraction procedure for removing the corrosion inhibitor from the fuel.

The corrosion inhibitors themselves are typically combinations of a fatty acid (C_{17} or C_{18}) dimer, a substance which has a molecular weight approximately one-half that of the dimer, and other, lower molecular weight substances such as ethylbenzenes and naphthalenes. When a substance like this is injected onto a GPC column the larger molecules are eluted first, followed by the smaller molecules. Thus, the fatty acid dimer peak is eluted first, followed by the substances of lower molecular weight. A typical fatty acid dimer is the dimer of linoleic acid which has 36 carbons and a formula weight of 562 grams.

There were 4 tasks assigned to be performed during the contract period:

1. Provide data on qualitative gel permeation chromatography and multi-column gel permeation chromatography of up to 10 corrosion inhibitors.
2. Provide data and report on technique to extract corrosion inhibitors from typical U.S. Navy JP-5 jet fuel.
3. Provide data on quantitative levels of two corrosion additives at levels between 2 and 30 parts per million in JP-5 jet fuel.
4. Provide report on the possible fate of corrosion inhibitors with time under varying real-world conditions, e.g., 1) seawater present, 2) effect of metal surfaces on corrosion inhibitor levels, 3) extraction of corrosion inhibitors added to two JP-5 fuels, 4) corrosion inhibitor levels in one or two "real" fuels.

EQUIPMENT

Samples were subjected to GPC analysis on a system which used a modular Spectra-Physics pump system with a Waters Model 401 differential refractive index detector. The pump system utilized a Model 740 dual-piston reciprocating pump, a Model 740-C pump control and a Model 714 pressure monitor. Samples were injected into a Rheodyne Model 7125 loop/valve - type injector. Chromatograms were recorded using either a Varian Model 9176-07 or a Fisher Recordall Series 5000, Model D5117-5AQ strip - chart recorder. For some of the GPC peaks integration was performed using a Hewlett Packard Model 3390-A integrator. Fisher HPLC grade uninhibited tetrahydrofuran (THF) was used as the mobile phase. Columns are described in the experimental sections.

TASK 1

Experimental

Nine corrosion inhibitors were supplied by their respective manufacturers. Samples of each were dissolved in THF to concentrations of 1000 ppm (V/V) and, subsequently, injected onto a GPC column for separation. Initial work was done using a Beckman-Altex u-Spherogel column (Model 255-81; 100 Å pore size) but it was found that slightly better separation could be achieved through the use of a 50 Å column (Model 255-80). Therefore, the 50 Å column was used for most subsequent separations. Later work was done using several columns in tandem in an effort to enhance separation. Several combinations were used: 2-50 Å columns; 2-50Å + 100 Å; 2-50 Å + 2-100 Å columns. The typical injection volume was 100 µl and, in general, flow rates (mobile phase) were 1 ml/min.

Results

Figures 1-5 are representative GPC chromatograms obtained for the 9 corrosion inhibitors available for study. These were obtained using a single 50 Å column. Chromatograms are read from left to right; the heaviest (left most peak) component corresponds to the fatty acid dimer. It can be seen that there are distinct similarities and differences between the graphical GPC data of the 9 inhibitors; each chromatogram exhibits a peak that corresponds with a substance of molecular weight greater than 500 - the dimer. This peak, in each case, has a retention volume of approximately 6 ml (3 cm from the injection point). See Table 1. Each chromatogram also exhibits one or more large peaks which begin with an elution volume of 8 ml (4 cm). These peaks represent

substances of much lower molecular weight, probably ethylbenzenes and naphthalenes, that may well act as a solvent system for the active ingredients. It can be seen that the ratio of the first (and major) of these low molecular weight peaks and the fatty acid peak varies considerably from additive to additive. A major difference, other than the peak ratios, to be observed from chromatogram to chromatogram is a second peak, possibly an acid phosphate ester (2) which follows the fatty acid peak in all GPC spectra except that of Unisor J. In some cases (NALCO 5403, HITEC 580) it appears as a poorly resolved shoulder on the trailing side of the dimer peak. Elsewhere it appears to be a well-resolved peak, in some cases of greater intensity than the fatty acid peak. While the retention volume is, for most of them, about 0.5ml greater than that of the dimer, (Table 1) there are two exceptions; DCI-4A and Mobilad F-800 show 0.9ml differences between the first and second resolved peaks. The du Pont additive, however, also has a shoulder on the trailing edge of peak 1, the retention volume of which resembles peak 2 in most of the other additives.

Of particular note is the relative intensity of the dimer peak from additive to additive for constant (1000 ppm (V/V) concentrations. There is as much as a 3-fold difference in peak height (compare UNICOR J with TOLAD 249) for similar V/V concentrations. A change to W/V calculations would make little difference because of similarities in the calculated densities of the additives. The peak height, and thus peak area differences could make quantitation difficult unless it could be known which additive was added to a particular fuel or unless the chromatograms could identify specific additives. Meanwhile, on a qualitative basis, it is fairly obvious that, in the absence of interfering species, different additives can be specifically identified on the basis of their detailed GPC chromatograms.

Samples were also run through several multi-column separation procedures in the hope that better resolution, particularly of the first two peaks, could be obtained. Results indicated little useful difference was made by the use of columns in tandem. The only real difference was the expected increase in retention volume and hence retention time. Thus, for this work, it was decided to use a single 50 A column. Future work may involve the use of a multi-column system. However, it is likely that other system modifications will need to be made in order to use the multi-column capability to best advantage.

TASKS 2, 3

Experimental

Corrosion inhibitors were extracted from fuels using a method similar to that used by Hillman et.al. (3) There are, however, differences in the relative volumes of solutions used and in the extracting solutions themselves.

Our method uses the following procedure: A volume of jet fuel is first extracted with an equal volume of 0.2M NaOH. For our purposes we use 250 ml of jet fuel and 250 ml of aqueous base. The two phases are shaken well together and allowed to separate. The aqueous phase is then drawn off and acidified with concentrated HCl to pH 2, which is well below pKa for the organic acids. After acidification the aqueous phase is back extracted with an equal volume of methylene chloride and allowed to evaporate to dryness at ambient temperature. The material left in the beaker after evaporation is

dissolved in small portions of THF, the inside of the beaker is rinsed well and the dissolved material is collected in a corner of the beaker. It is eventually taken up in exactly 5.0 ml of THF and transferred to a glass vial with a teflon cap liner.

For much of this work commercial aircraft fuel, Jet A, was used as the base fuel. Jet A was chosen because it is similar to JP-5 and contains no mandatory additives. Samples of Jet A were spiked with known quantities of the additives used to make solutions which had known concentrations of corrosion inhibitors in jet fuels. The additives chosen for study were Unicor J and Mobilad F-800. To prepare samples for extraction and subsequent GPC analysis corrosion inhibitors were added to the Jet A by syringe and the fuel was then shaken well to dissolve the corrosion inhibitor. Samples prepared to test the method were made up in the concentration range 2-30 ppm (V/V).

It should be noted here that density determinations were made for each of the additives. In general, the densities were in the range 0.92-0.94 g/ml. Thus, a sample which was 10 ppm (V/V) would be approximately 9.2 ppm (W/V). Comparator standards of known concentration were prepared by dissolving calculated quantities of corrosion inhibitor in appropriate volumes of THF.

The system used has been described elsewhere in this report. For most of the experimental work, however, the following parameters were used:

- | | |
|--------------------|------------|
| (a) flow rate | 1 ml/min |
| (b) chart speed | 0.5 cm/min |
| (c) recorder range | 10 mV |

- (d) injection volume 80-100 ul
- (e) detector span x4
- (f) temperature ambient
- (g) solvent THF
- (h) integrator time 0.64 msec
constant
- (i) integration threshold 2 exp 4

Data for many of the experimental runs were recorded with the HP 3390-A integrator. All peak areas were obtained through the use of this instrument. Peak height and retention volume information were also reported through the use of the integrator.

Samples were drawn from their respective vials and injected onto the column via the injector. A 200 ul maximum capacity sample loop was used. When extracted samples were run for analysis, comparator standards of approximately the same concentrations were run in the same experimental period. Thus, direct concentration comparisons were possible. In general, the comparators were freshly made on the day of the GPC analysis.

Results

Initial work performed on the development of the extraction method used dissimilar volumes of fuel and aqueous base followed by the acidified aqueous phase/methylene chloride extractions. There were typically three extractions performed in each phase of the process and the volumes were combined. The extraction efficiency for samples extracted in this manner

was found to be no better than 88% as determined by GPC analysis and comparison of peak heights and areas with comparators. Parameters such as pH of the aqueous phase and recovery of the additive after evaporation of the methylene chloride phase were adjusted with varying results. However, none yielded 100% recovery of the additive.

A series of extractions was performed to determine optimal extraction volume parameters. Each extraction involved 250 ml of Jet A spiked with 50 ppm (V/V) of Mobilad F-800. Results are shown on Table 1. For the first set of extractions, the volume of aqueous phase was varied and the volume of methylene chloride used to perform each back extraction was equal to the volume of aqueous phase used. Thus, the Jet A sample extracted with 175 ml of aqueous base used a back extraction volume of 175 ml. A second and third series of extractions which used extraction volumes of aqueous and organic phases equal to those used in the first series were also run on the same Jet A samples. GPC analysis of these fractions indicated that extraction was complete for the 250 ml/250 ml system after the first extraction, whereas second and third series extractions on the 250 ml/175 ml and 250 ml/100 ml systems yielded detectable quantities of Mobilad F-800. For the second set of extractions the volume of aqueous base used was held constant at 250 ml and the volume of methylene chloride used to back extract the samples was varied. It can be seen, from Table 2, that extraction with equal volumes of fuel/aqueous base followed by a back extraction which uses the same volume parameters provides optimum efficiency.

Only one extraction series was performed on this second set of samples. Quantitation was accomplished by comparing areas of the sample peaks with that of a comparator made to have the same concentration in THF as the theoretical concentration of an extracted sample of 50 ppm Mobilad F-800. It can be seen that extraction efficiency is optimized when the volumes of extracting solutions, whether for the direct or back extraction, are equal to that of the jet fuel. For those extractions all the Mobilad F-800 added to the fuel was recovered.

To test the method 250 ml samples of Jet A were spiked with enough Mobilad F-800 to make samples which were 5, 10, 15, 20 and 30 ppm (V/V) in the additive. Each sample was then extracted with one 250 ml volume of aqueous base and re-extracted with one 250 ml volume of methylene chloride following acidification of the aqueous phase. After extraction the additive was recovered as previously described and then taken up in 5 ml of THF. Comparator standards were made by dissolving the same quantity of additive as had been used to spike the jet fuel samples in 5 ml portions of THF. One comparator was made for each spiked sample. The same procedure was used for an identical series in which the additive used was Unicor J. Figures 6 and 7 are plots of GPC peak area versus concentration for the Mobilad F-800 and Unicor J series respectively. Both plots include points for the extracted and standard (comparator) samples. It is obvious from the data obtained and plotted that the sample and standard curves coincide exactly, attesting to the efficiency of the method.

Task 3 requires data on corrosion inhibitor concentrations as low as 2ppm (W/V). Our work indicates that attaining this concentration level is feasible. However, there are some difficulties which derive largely from the fact that there are components of the jet fuel itself which extract into the aqueous base and are carried over into the final sample along with the corrosion inhibitor. Figure 8 is a gel permeation chromatogram of a sample of additive - free Jet A which has been carried through the extraction process. The first peak appears at a retention volume of approximately 6.8 ml and can interfere with or mask a low - intensity additive peak. Thus, in order to obtain meaningful quantitative results, a procedure should be developed which would exclude the fuel contribution to the GPC results. Meanwhile, low concentrations (2 ppm) can be semi-quantitatively determined by estimating the peak height of the additive peak which appears as a shoulder on the leading edge of the fuel component peak.

Task 4

Experimental

Task 4 had four parts, two of which dealt with the possible fate of corrosion inhibitors and two of which dealt with the extraction of corrosion inhibitors from JP-5 "real world" fuels. For the parts of Task 4 which dealt with the fate of inhibitors two experiments were performed. One tested the "plating out" theory - that is, that the inhibitor tends to plate out on the metal surfaces of transfer lines and

storage tanks. A second experiment was performed to determine whether there is a "ionic complexing" effect when the fuel, and hence the inhibitor, is in contact with seawater. The plating out experiments measured inhibitor loss as functions of time and metal surface area.

In the first experimental set, extractions were performed on a set of six 250 ml samples of a JP-5 focus fuel (NRL#85-12) each of which had been in contact with a constant weight (approximately 19 g/sample) and hence a relatively constant surface area (estimated to be about 400 cm^2), of clean stainless steel wool. Samples were subjected to varying periods of contact time with the steel wool. For the second experimental set (Jet A was used as the fuel) the mass (and thus the surface area) was varied and time was held constant. All samples removed for extraction were 250 ml and additive (Mobilad F-800) concentrations were 20 ppm (V/V). Extraction and GPC procedures used were the same as those described elsewhere.

The "ion complex" effect was tested as follows: a 750 ml sample of Jet A was spiked with 15 microliters of Mobilad F-800 to yield a solution with an additive concentration of 20 ppm (V/V). A 250 ml aliquot (sample 1) was removed and placed in a clean bottle and stored away from light for 16 days. A second 250 ml aliquot (sample 2) was removed and placed in a bottle which contained 250 ml of (synthetic) seawater and subjected to the same storage conditions. Both bottles were occasionally gently agitated, but not shaken, to simulate the rolling motion of a ship.

Finally the last aliquot (sample 3) was immediately extracted with seawater. After the 16 day storage period, sample 1 was extracted with aqueous base and the seawater layer from sample 2 was separated from the fuel. The aqueous layer, whether seawater or base, from each sample was acidified to pH2 and the usual procedure for back - extraction and GPC analysis was followed. In a later experiment, spiked fuel samples were exposed to distilled water rather than seawater to test the "ion complex" effect.

In the second part of this task, 2 samples of additive-free JP-5 fuel were spiked with additive, then extracted and subjected to GPC analysis. Samples used for this experiment included NRL#85-11 and 85-12 These samples were spiked prior to extraction and analysis with 20 ppm (V/V) of Mobilad F-800.

As part of this experimental set, a "real world" sample of JP-5 fuel (#83-85) was obtained from Andrews AFB for analysis of corrosion inhibitor present. No additional corrosion inhibitor was added. The sample was extracted and subjected to GPC analysis following the procedure established.

Results and Discussion

Effect of Metal Surfaces: Table 2 summarizes the results of the time/concentration and surface area/concentration studies. For the time/concentration studies it appears likely that there is no significant concentration decrease over the long term. It is probable that there is an almost immediate plating out (1.5 hours) followed by a period of relatively little activity. The differences in the per cent recovery data between the first 3 and last 3 samples could be due to variations in the steel wool surface area. An average of all 6 results yields 73.7% recovery. The control sample (#7) was treated the same way as samples 1-6 with the exception that there was no steel wool present in the beaker used to age the samples. It is likely that incomplete recovery of the additive is caused by adsorption (plating-out) of the inhibitor on the glass surface. At the 5 ppm level of acid dimer estimated in these samples, a monolayer surface area coverage would be about 2000 cm^2 . This means that about 11% of the acid dimer would be expected to plate out on the beaker walls and an additional 20% would plate out on the steel wool surface.

For the surface area/concentration studies, the data are in substantial agreement with those obtained in the time/concentration study. It is possible that one would need more precise control of the surface area than that provided by steel wool to show precise differences in the concentration of additive recovered. It is also possible that other surfaces should be examined. In any case, this work seems to indicate that the "plating out" effect of corrosion inhibitors on metal and glass surfaces plays a definite part in additive loss. Additional experiments on other surfaces are currently underway.

Effect of Seawater:

The seawater experiments yielded truly interesting results. Sample 1, the control sample, was extracted with aqueous base after the 16 day storage and analyzed, using freshly prepared comparators, for Mobilad F-800. The per cent recovery was found to be 83.2% which is quite consistent with results obtained from the previous set of experiments for additive adsorption (plating-out) on inside surfaces of storage containers.

Sample 2, which was stored over seawater, was extracted with base and analyzed using our method. No additive was recovered. The seawater layer was acidified, back-extracted and subjected to GPC analysis with identical results. An amber-colored solid was observed to form at the interface between the fuel and seawater. This insoluble material was isolated from the system. It was dissolved in acid to pH 2, extracted with methylene chloride and analyzed. The results were not quantitated, however GPC analysis yielded a substantial additive peak. There is about 375 mg of Ca^{++} and Mg^{++} available in 250 ml of seawater. This is more than adequate to effect an insoluble interfacial salt formation with the 5 mg of dimer acid in 250ml of fuel.

Sample 3 was extracted immediately (no storage period) with seawater and then subjected to the same procedure as was applied to Sample 2. Exactly the same results were obtained; that is, that the additive was found only in the interfacial material. The obvious implication from these experiments is that there is a salt formation between the additive and the Group II metal ions and that the soap formed is insoluble in both liquids. Thus, seawater intrusion into fuel storage tanks would be likely to remove any corrosion inhibitor present.

A series of experiments which substituted fresh (distilled) water for seawater produced no interfacial material and analysis of the aqueous layer yielded no additive peak. Thus it can be concluded that water itself is not involved in the reduction of additive concentration.

Analysis of Spiked JP-5 Fuels: 250 ml samples of additive free JP-5 fuels 85-11 and 85-12 were spiked with 20 ppm (V/V) of Mobilad F-800. The extraction/GPC procedure already described was applied to each and 100 per cent of the additive was recovered from sample 85-11. Sample 85-12 yielded a recovery of 95 per cent. GPC additive peaks were compared with standards and the results were based on peak heights. We have found that peak height results are sometimes low by as much as 7-8 per cent. This work indicates that the procedure we have developed for extraction and determination of corrosion inhibitors in jet fuels is well suited to JP-5 fuels.

Analysis of JP-5 Sample 83-85: 250 ml of this fuel was analyzed following our procedure. Because the additive manufacturer was not known it was not possible to provide information which is better than semi-quantitative. If the additive were DII-4A, Mobilad F-800 or Unicor J, we would estimate the concentration to be in the range of 5-7 ppm. However, if some other additive was used, the concentration could be as high as 12-15 ppm.

Work must be undertaken to qualitate the corrosion inhibitors if truly quantitative results are desired. If, however, only minimum concentration values are needed or if approximate concentration ranges would suffice, less emphasis needs to be placed on identification of the corrosion inhibitor present.

Summary and Conclusions

A method has been established for quantifying corrosion inhibitor levels in Navy Jet fuels and 100 per cent recovery of the additive from fuel is possible. Concentrations of 5 ppm (V/V) are easily determined and the detection and determination of as little as 1-2 ppm is possible. Work should be done, however, to qualitate the additives so that proper comparators can be made and results optimized.

Results obtained from studies dealing with the fate of the corrosion inhibitors are interesting and indicate that seawater, in particular, effectively removes the corrosion inhibitor from fuel. The need to expand this work to include more studies on the "plating-out" or adsorption phenomena is indicated.

References:

1. Navy Mobility Fuels Evaluation, Final Report, July 20, 1984, Contract No. N00014-82-C-2370 prepared by the Chemistry Laboratory, TRW Energy Development Group, for the Naval Research Laboratory, Washington, D.C.
2. Blok, A.P., Dahmen, E.A.M.F. and Verjaal, A.J., J. Inst. Pet., 51, 502 pp. 348 - 353 (October 1965).
3. Hillman, D.E., Paul, J.I. and Cobbold, D.G., "Determination of Pipeline Corrosion Inhibitor (Hitec E 515) in Aviation Fuels by Gel Permeation Chromatography" from Recent Developments in the Petroleum Industry, ed. Hodges, D.R., Appl. Sci. Publ. Ltd. (1977).

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Table 1 Qualitative GPC Analysis of Approved Military Specification
Corrosion Inhibitors

<u>Additive</u>	<u>Manufacturer</u>	<u>Retention Volume(ml) *</u>	
		<u>Peak 1</u>	<u>Peak 2</u>
DCI - 4A	E.I. duPont deNemours & Co. (Inc.)	6.0	6.9 ^a
NALCO 5403	Nalco Chemical Co.	6.0	6.5
NALCO 5403	Nalco Chemical Co.	6.0	6.5
HITEC 580	Ethyl Corp.	5.8	6.3
TOLAD T-245	Petrolite Corp.		
TOLAD T-249	Petrolite Corp.	6.0	6.6
UNICOR J	Universal Oil Projects	5.9	---
MOBILAD F-800	Mobil Chemical Co.	5.9	6.8
LUBRIZOL 541	Lubrizol Corp.	5.9	6.4

* Data taken from Figures 1-5; peaks are in order of elution

^a Well-resolved peak

Table 2 Results of Extraction Series Performed to Determine Optimum
Extraction Volume Parameters

<u>Sample, Extraction Series</u>	<u>V_{NaOH}/V_{MeCl_2}</u>	<u>Area of Dimer Peak (integrator counts)$\times 10^5$</u>	<u>Extraction Efficiency %</u>
1	250ml/250ml	4.01	102
1	175ml/175ml	3.62	92
1	100ml/100ml	3.35	85
2	250ml/250ml	4.07	103
2	250ml/175ml	3.62	92
2	250ml/100ml	3.55	90

Average area of comparator standard peak (integrator counts)
 $= 3.92 \pm 0.05 \times 10^5$

* 4 injections were made

Table 3. Results of "Plating - Out" Experiments

A. Time/Concentration Study

<u>Sample</u>	<u>Exposure time (hrs)</u>	<u>Per cent of Additive Recovered</u>
1	1.5	72.5
2	3.0	72.8
3	20	74.1
4	93	58.1
5	168	83.3
6	280	81.0
7(Control)	280	88.6

B. Surface Area / Concentration Study

<u>Sample</u>	<u>Grams Steel Wool</u>	<u>Per cent of Additive Recovered</u>
1	7.5	83.5
2	15.0	73.8
3	22.5	73.8
4	30.0	71.6

Legend of Figures

- Figure 1. Typical gel permeation chromatograms of Mobil-F800 and Lubrizol 541 dissolved in THF (1000 ppm w/w). RI detector, THF flow of 1 ml/min.
- Figure 2. Typical gel permeation chromatograms of DCI-4A and Nalco 5403 dissolved in THF (1000 ppm w/w). RI detector, THF flow of 1 ml/min.
- Figure 3. Typical gel permeation chromatograms of Nalco 5405 and Hitech-580 dissolved in THF (1000 ppm w/w). RI detector, THF flow of 1 ml/min.
- Figure 4. Typical gel permeation chromatograms of Tolad T-249 and Unicor J dissolved in THF (1000 ppm w/w). RI detector, THF flow of 1 ml/min.
- Figure 5. Typical gel permeation chromatograms of Tolad T-245 dissolved in THF. RI detector, THF flow of 1 ml/min.
- Figure 6. Plot of extracted and standard concentrations of Mobilad F-800 versus peak area of active ingredients.
- Figure 7. Plot of extracted and standard concentrations of Unicor J versus peak area of active ingredients.
- Figure 8. Typical gel permeation chromatograms of additive-free Jet A after extraction. Detector and flow conditions as in Figures 1-5.

MOBIL-F800

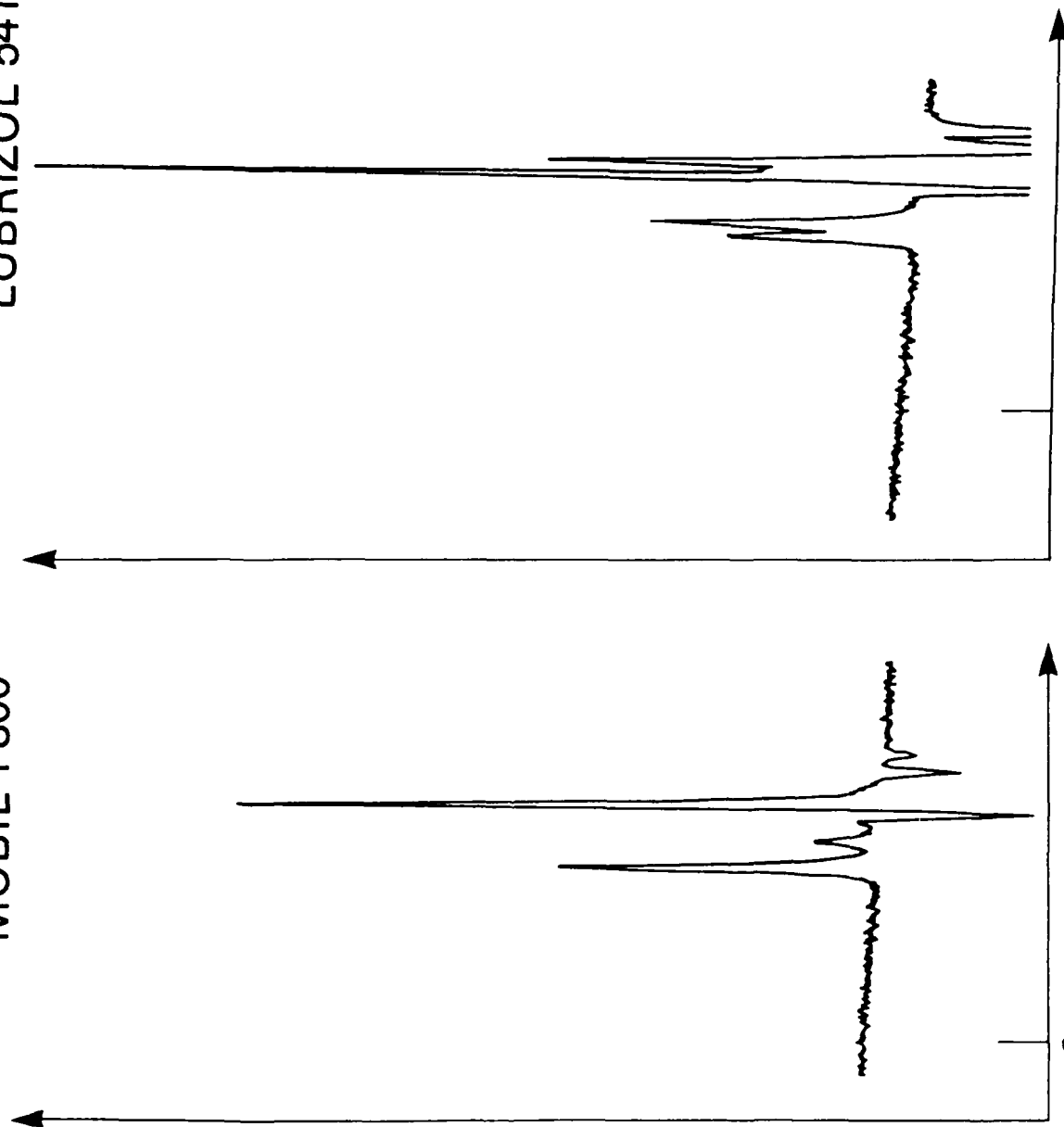
LUBRIZOL 541

RESPONSE (mV)

TIME (min)

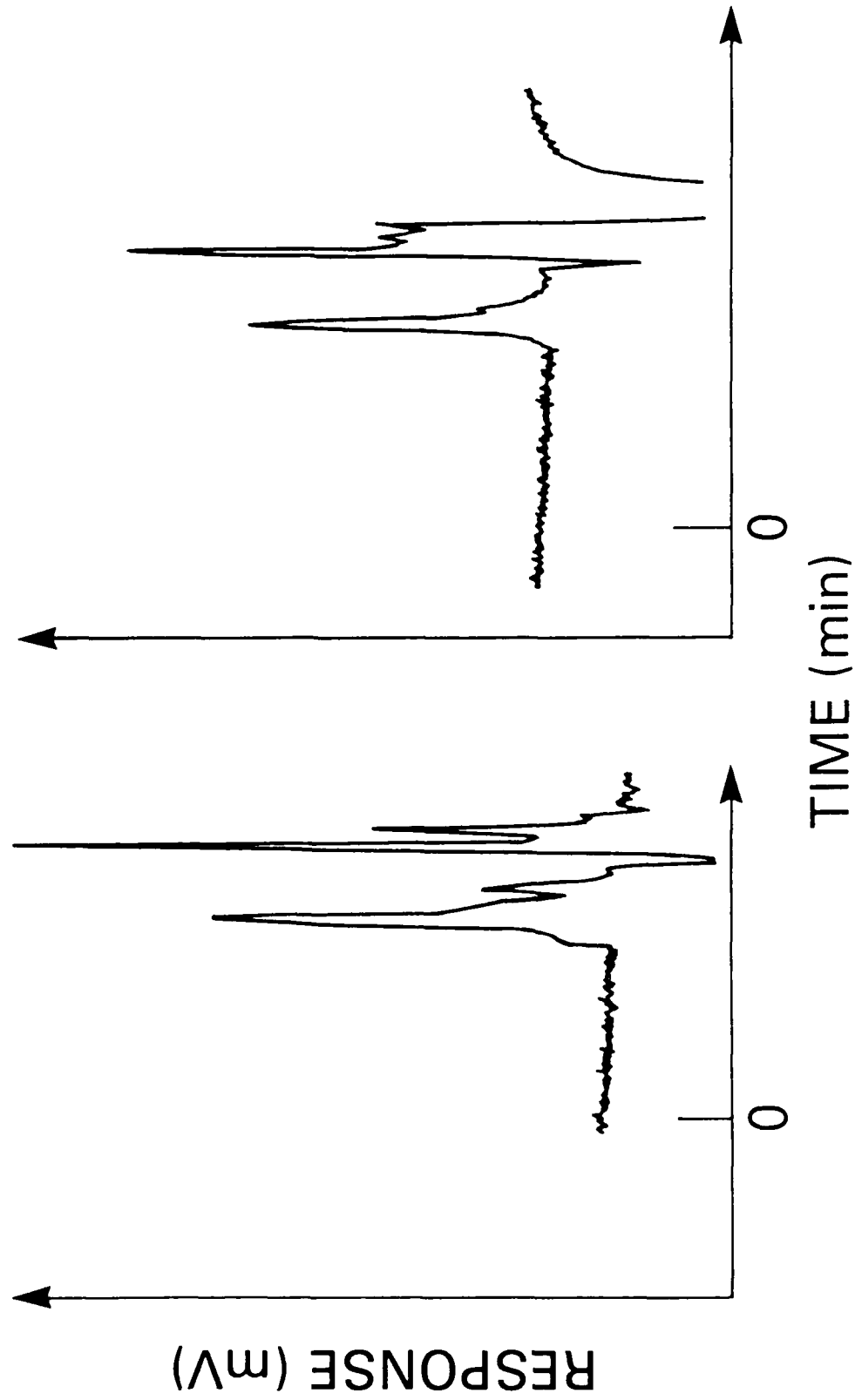
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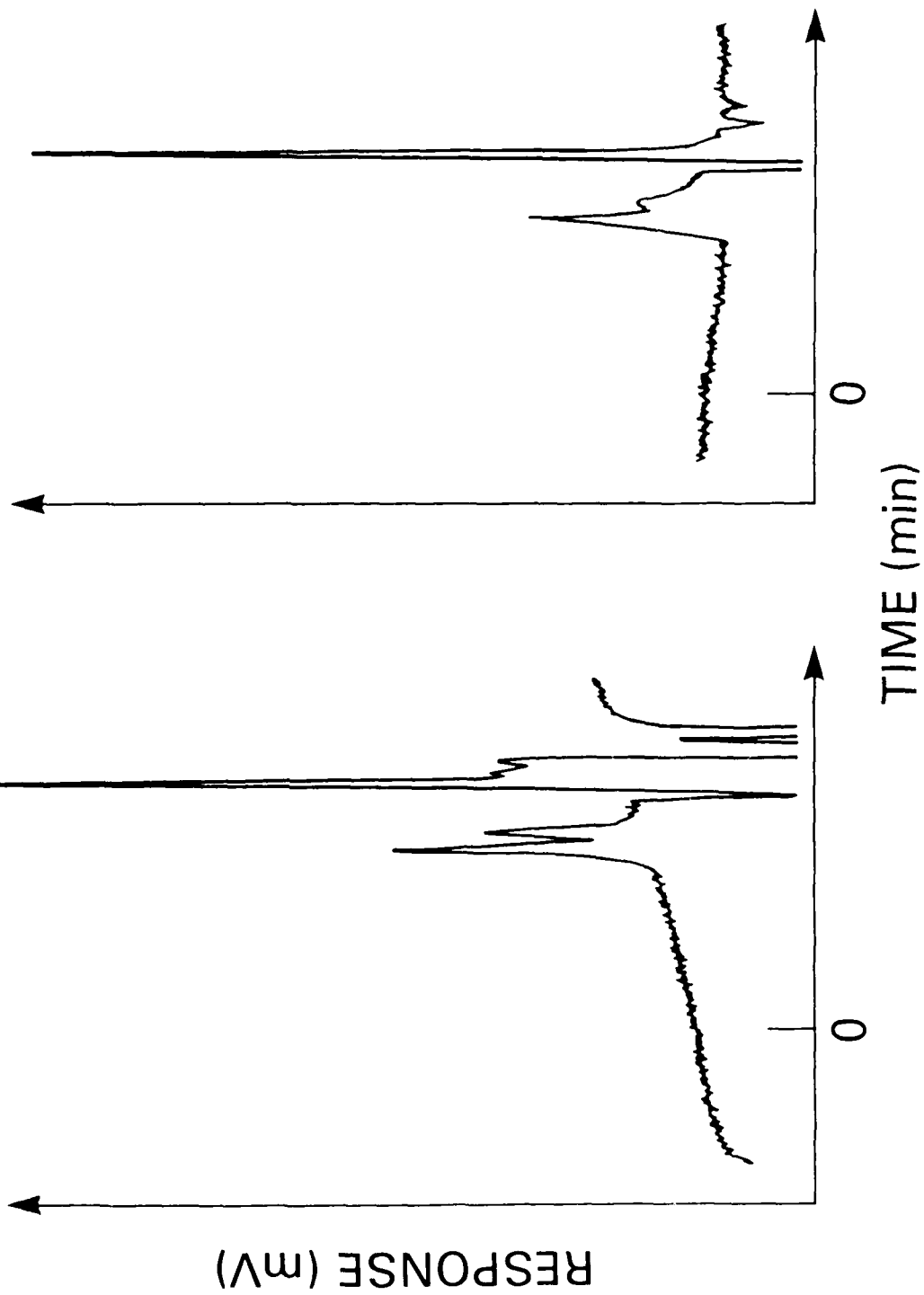
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NALCO 5403



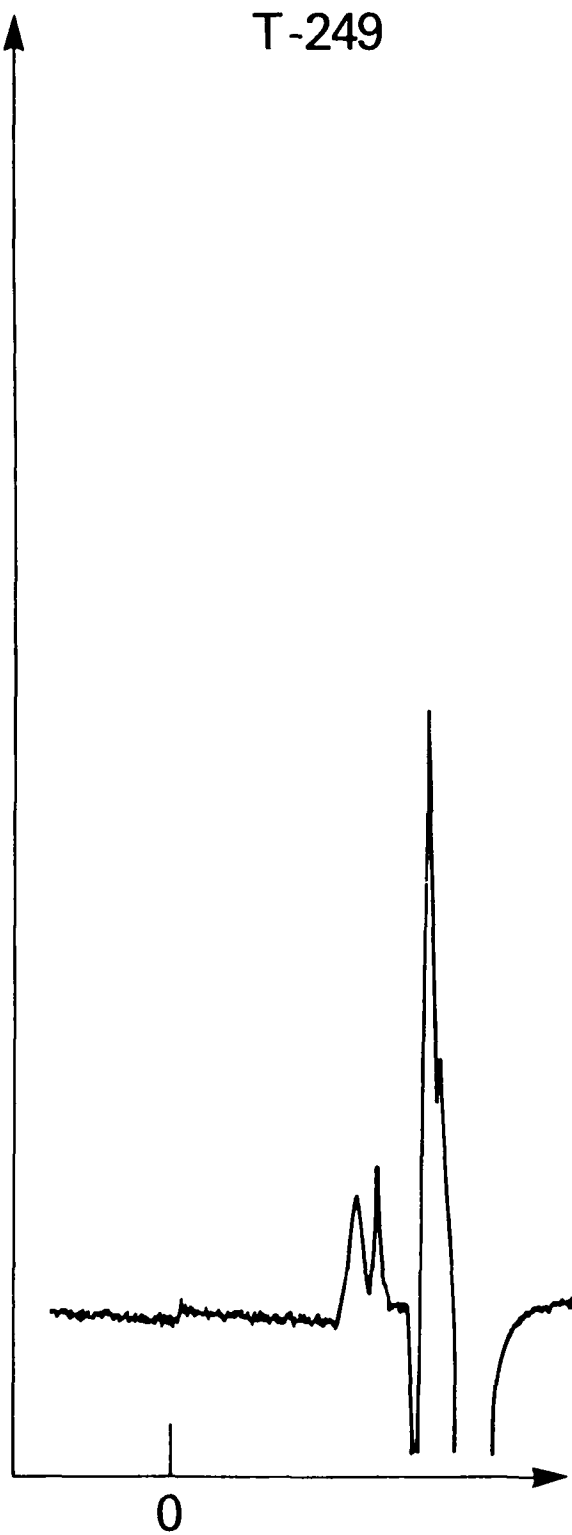
HITEC-580

NALCO 5405

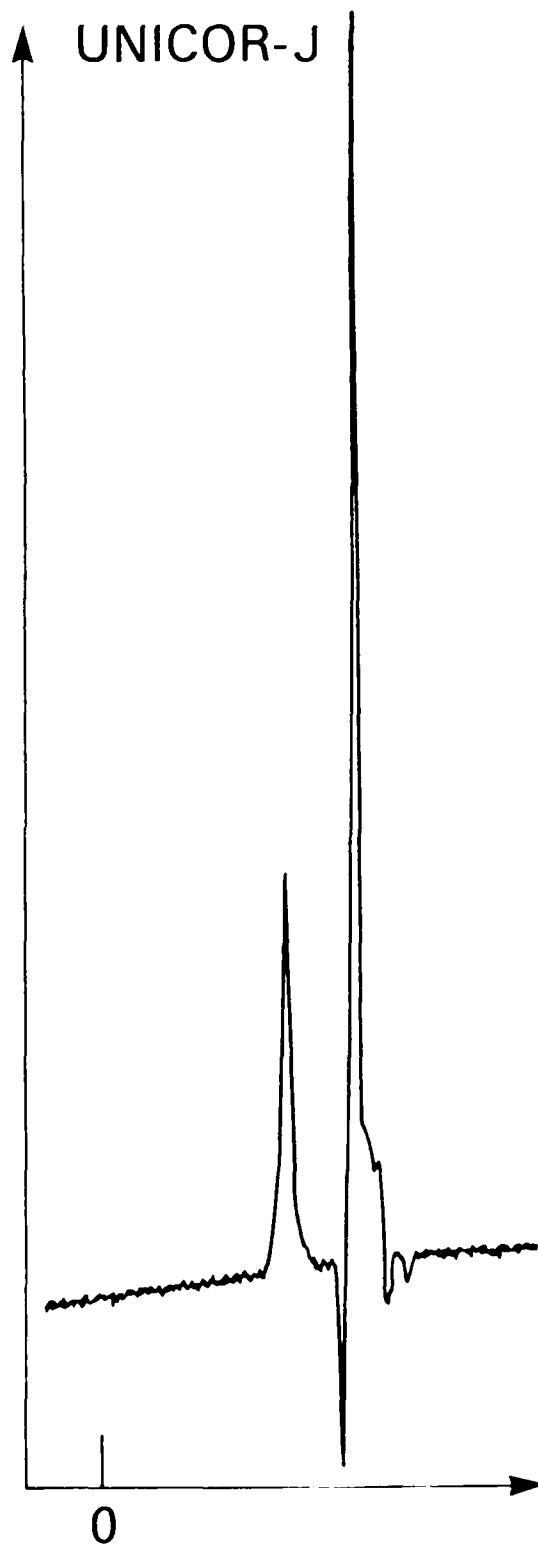


RESPONSE (mV)

T-249



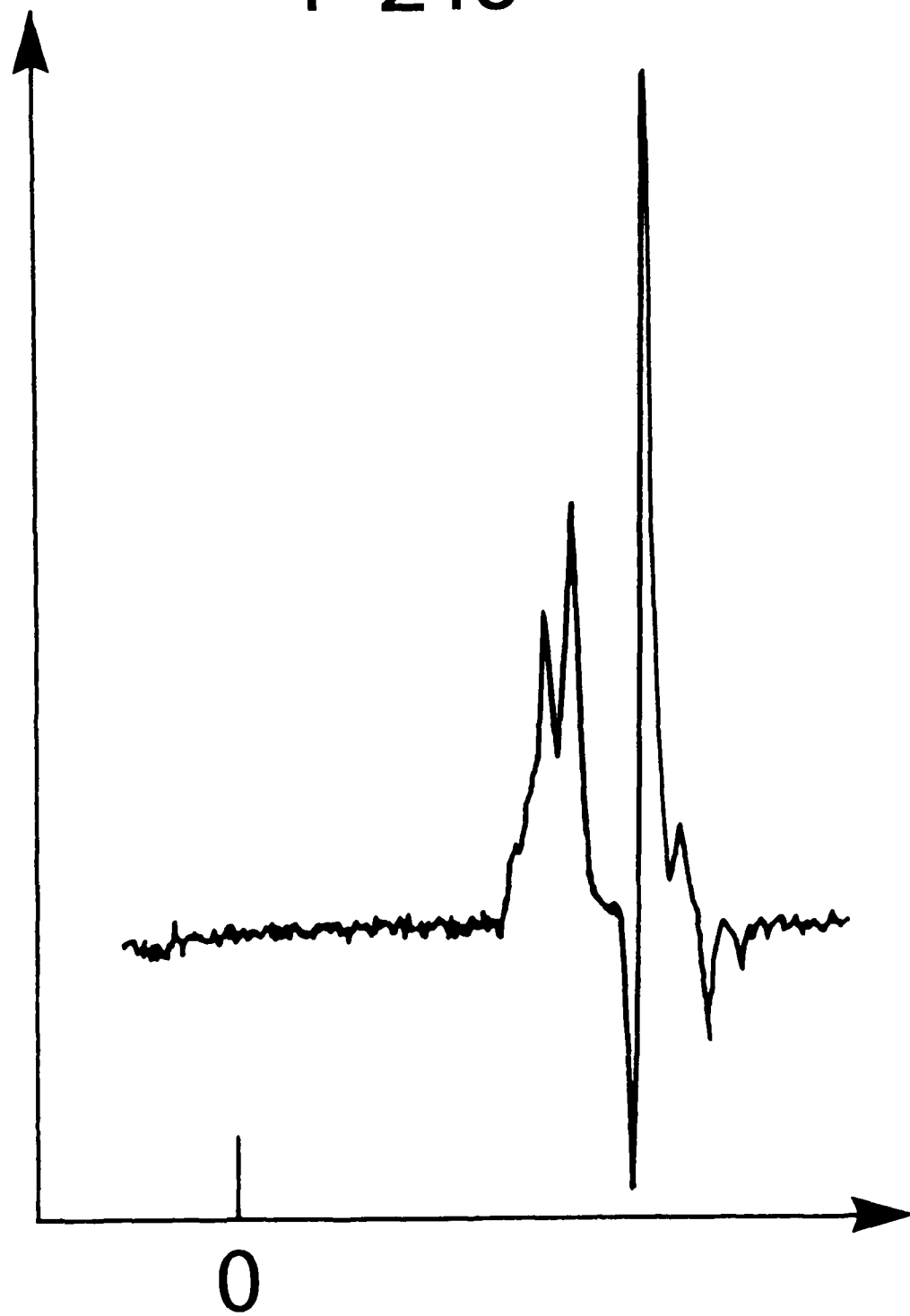
UNICOR-J



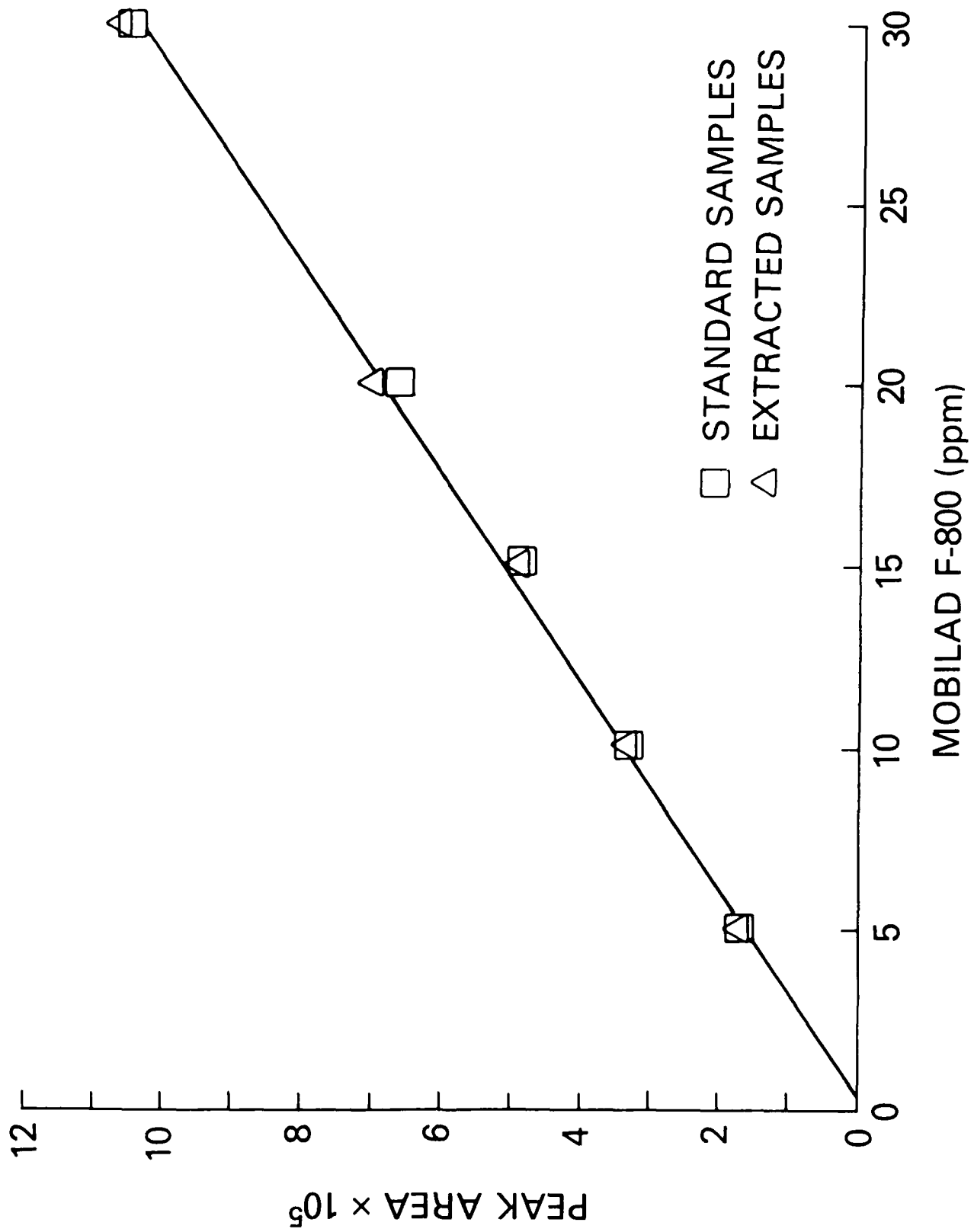
TIME (min)

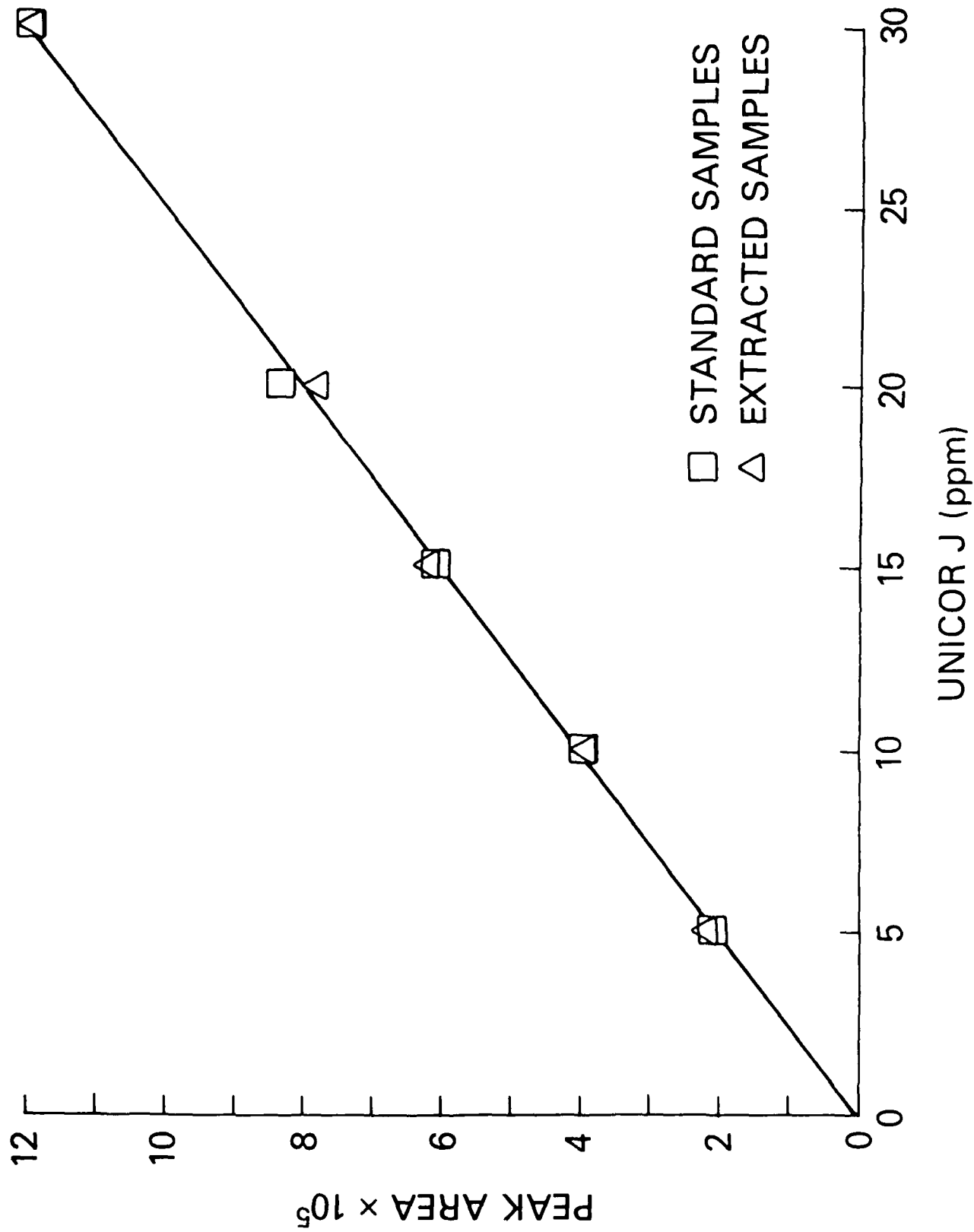
T-245

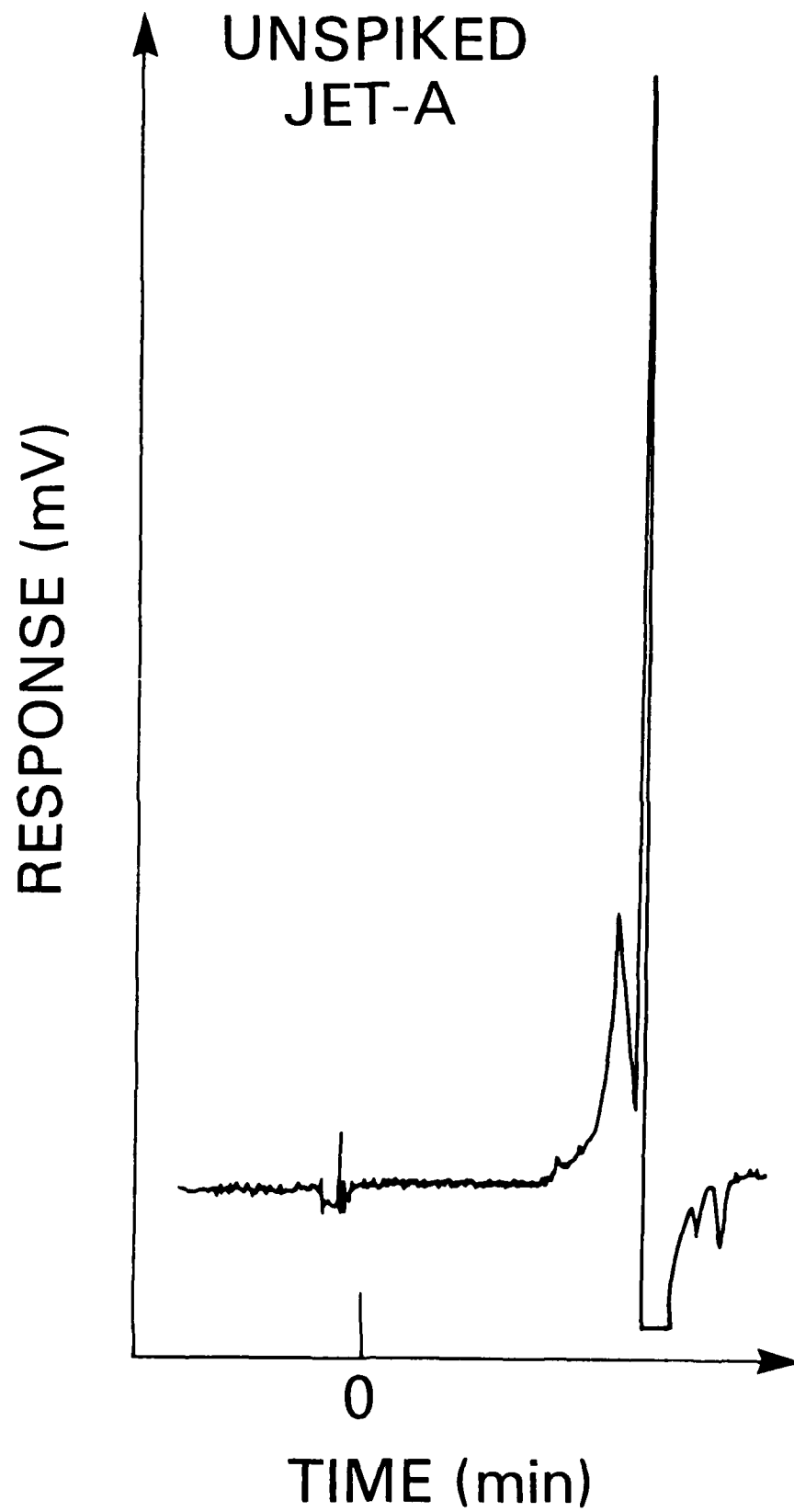
RESPONSE (mV)



TIME (min)







DTIC

FILMED

4-86

END